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(AFML-TR-78-19)

LEVEL II

(2)

EXPLORATORY DEVELOPMENT OF COATED FABRIC FOR FIRE PROXIMITY SUITS

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JUNE 1978



FINAL REPORT FOR PERIOD JUNE 1976-OCTOBER 1977

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**CIVIL AND ENVIRONMENTAL
ENGINEERING DEVELOPMENT OFFICE**

(AIR FORCE SYSTEMS COMMAND)

TYNDALL AIR FORCE BASE

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This technical report has been reviewed and is approved for publication.


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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An aluminum-colored, coated outer fabric for use in fire proximity suits has been developed. The basic fabric used was a filament Kevlar fabric weighing 6.7 oz/yd. This was primed with Viton L-31, followed by three coats of Viton A containing brown pigment, a polyurethane topcoat containing aluminum pigment, and a finish coat of clear polyurethane. The fabric does not burn, and exhibits good resistance to heat transfer. It is expected to be more comfortable and durable than the presently used aluminized Novatex fabric. Three hundred yards of the fabric were delivered to the Air Force for manufacture of experimental		

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fire proximity suits of conventional design, and 30 yards of manufacture of
six experimental suits of unconventional design.

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Preface

The work described in this report was performed by Fabric Research Laboratories (FRL), a division of Albany International Corporation, during the period April, 1976 to December, 1977. It was part of a larger development program being carried out at Tyndall AFB. Technical supervision was the responsibility of the Air Force Materials Laboratory, Wright-Patterson AFB, the Project Engineer being Mr S. Schulman. Advice and assistance of Major Birney T. Pease and Mr Norman D. Knowles, both of Tyndall AFB, is gratefully acknowledged.

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SECTION I

INTRODUCTION

In Contract F33615-74-C-5117, FRL developed for the Air Force a nonflammable, highly reflective coating for use as an alternative to the aluminized coating currently used in firefighters' proximity suits. This coating consisted of Viton, a fluorocarbon coating compound, filled with bronze powder to give high IR reflectance, and carrying a thin polyurethane topcoat for improved wear resistance. The substrate fabric used was made from asbestos-covered Nomex yarns by Raybestos-Manhattan, Inc. and known by their tradename of Novatex.

At the close of this contract, FRL coated and delivered to the Air Force sufficient quantity of this fabric for them to make up some experimental suits for wear testing. The bronze coated fabric was found to be more flexible than the aluminized fabric, and had nearly as high an initial IR reflectance. However, several faults were found with the fabric during the wear trials:

- 1) inadequate abrasion resistance - the coating had been worn off many of the protruding knuckles in the fabric weave
- 2) dirt adhesion due to the surface tackiness of the urethane topcoat
- 3) inadequate adhesion of the coating to the base fabric
- 4) yarn separation, particularly at the knees, due to slippage of yarns in the base fabric
- 5) The bronze colored surface was not sufficiently visible in a smoke or water vapor filled atmosphere, and the firefighter could not be seen as easily as with the aluminum-colored surface.

The present program was instituted to attempt to overcome these faults.

SECTION II

BASE FABRIC

All of the above faults except the dirt adhesion were felt to be related to the construction of the Novatex base fabric. The design was such that protruding knuckles at yarn cross-over points created sites for excessive abrasion. Coating adhesion was made difficult because of the nature of the yarn construction. The coating had to adhere to the asbestos covering on the yarns, and adhesion to asbestos is generally poor. Moreover, the cohesion between the asbestos fibers and the filament Nomex yarns which maintain the structural integrity was relatively poor. For these reasons, and others related to design flexibility and cost, it was decided not to use Novatex fabric in the new designs.

In addition to Novatex, we were to consider the use of 2 weights of 50/50 Kynol/Nomex, 2 weights of 70/30 Kynol/Nomex, staple HT-4, Durette, and 2 weights of staple Kevlar fabric. The following conclusions were reached, in conjunction with the Project Engineer:

1. 50/50 Kynol/Nomex

We decided to concentrate first on the 70/30 blend, and if this looked encouraging, to consider the 50/50 blend.

2. 70/30 Kynol/Nomex

Extensive trials were run using two fabric weights: #162 was a 10-ounce fabric and #1110 was a 6-ounce fabric. Although these fabrics accepted the coating well, it became clear as the work progressed that the agencies responsible for proximity suit redesign were interested in the possibilities of using Kevlar because of its high puncture resistance.

3. Staple HT-4

This was an experimental fiber which duPont decided not to manufacture. It is no longer available.

4. Durette

This was not pursued because of the decision to use Kevlar.

5. Staple Kevlar

At the outset of this contract, there was some doubt about the long-term availability of Kevlar, particularly in staple form. A discussion held between the Project Engineer, FRL and a representative of duPont convinced us that Kevlar could be expected to remain a commercial fiber, though staple Kevlar, at that time, was still available only in limited supply. It was decided, on the basis of this discussion, not to proceed with the use of staple Kevlar, but to substitute a filament Kevlar fabric. Two suitable fabrics were listed as stock items by J. P. Stevens, Inc. They were as described in Table 1.

Also included in this table are values for the characteristics of some hand coated samples using a clear Viton tiecoat, 2 coats of bronze pigmented Viton, and an aluminum pigmented acrylic topcoat, and for comparison the currently used aluminized Novatex.

On the basis of these results, it was decided to select Style 718 Kevlar 29 fabric for the substrate material because of its lower cost. Several experiments reported herein used Style 1110. However, the conclusions drawn from those experiments would apply equally to Style 718.

TABLE 1. PROPERTIES OF KEVLAR SUBSTRATE FABRICS

	Style 1110		Style 718		Alumi- nized Novatex
	Uncoated	Coated	Uncoated	Coated	
Yarn Denier	1000	---	1500	---	---
Ends x picks per inch	22 x 22	---	17 x 17	---	---
Weight (oz/yd ²)	5.8	9.2	6.8	10.6	14.4
Thickness (mil)	13	13	15	17	31
Stiffness (mg/cm ² /cm)					
warp	2400	10500	3700	18500	---
filling	1600	10400	1700	11700	20400
Warp Strength (lb/inch)	680	780	700	860	---
Tongue Tear (lb)	>150*	>150*	>100*	350	---
Heat Transfer (°C)					
20 sec, 1.3 cal/cm ² /sec	---	138	---	135	122
1977 Cost (\$/running yard)	\$11.05	---	\$7.95	---	---
	(60" width)		(50" width)		

*Load at which yarn slippage occurred. No tearing took place.

Prime Coat

DuPont has made available an aqueous fluoroelastomer dispersion, Viton L-31. We found that the L-31 made both an excellent fabric prime and back coat. By proper formulation and curing, adhesion of subsequent coatings to the fabric was improved and the flexibility of the coated fabric was enhanced because of little penetration of coating into the fabric. Table 2 lists the technical data on the Viton L-31 latex as well as the prime coat formulation and cure schedule.

TABLE 2. VITON L-31 LATEX

Solids Content (61-65%)	63% Specific
Gravity at 25°C (approximate)	
Latex	1.41
Polymer	1.86
Initial pH (declines with age)	6
Viscosity (cps) [Brookfield, 30 rpm]	150
Surface Tension (dynes/cm)	39.5
Emulsifier	anionic
Color	off-white to white
Odor (latex)	mild
Polymer soluble in low molecular weight ketone and esters	THF, DMF
Decomposition	above 288°C, nonflammable

<u>Coating Formulation</u>	<u>Parts</u>	<u>Wet</u>	<u>Dry</u>
Viton L-31		159	100
ZnO Dispersion*		20	10
Diak #3 Dispersion*		9.1	3
Dry coating at 95°C			
Cure at 175°C			

*Must be ball milled and properly dispersed.

Coating Pigment

The need for good visibility as well as high IR reflectance creates a serious problem. A bronze pigment that gave excellent IR reflectance and was compatible with the Viton elastomer was found to be unacceptable in terms of visibility in smoke and flame.

A number of alternatives to the bronze pigment was considered for evaluation. Two of interest are (a) Nickel Silver pigment powder O. N. Both Corp. #B9063; and (b) Stainless Steel pigment powder, U.S. Bronze Powders, Inc., #304-2.

Both pigments are compatible with Viton A but the IR reflectance of coated samples prepared using these pigments was unsatisfactory. In an effort to enhance IR reflectance, a bronze subcoat was used and a topcoat of Nickel-Silver pigment was tried using the following construction:

base fabric - #1110 Kynol/Nomex
L-31 prime coat - 1/3 mil
3 coats bronze pigment in Viton A
(1/1 bronze to Viton A) - 1.2 mil total
1 coat Desoto polyurethane and pigment (1/1) - 1/2 mil.

A visual improvement in reflectance did occur, but the IR reflectance was still unacceptable. At this point the same construction as above, but having a topcoat of Desoto polyurethane and aluminum pigment (#2011, O. A. Both Corp.) was made and tested for flammability. It was expected that the aluminum might violently react with the Viton, but it did not. In fact, the polyurethane topcoat was self-extinguishing once the flame was removed, and there was no progressive burning at all. It was concluded that a single topcoat of urethane with aluminum pigment did not create a flammable combination with Viton and offers excellent IR reflectance with reasonable visibility.

Table 3 lists the IR reflectance of the various pigment combinations referred to above.

TABLE 3. IR REFLECTANCE USING VARIOUS PIGMENT COMBINATIONS

Substrate: #1110 Kynol/Nomex, 6 oz with L-31 prime coat
Instrument: Beckman DK2 with integrating sphere.

<u>Viton Base Pigment</u>	<u>Topcoat Pigment</u>	<u>IR Reflectance (%)</u> *
Nickel-Silver	Nickel-Silver	65
BRPG Bronze	Nickel-Silver	68
Stainless Steel	Stainless Steel	48
BRPG Bronze	#2011 Aluminum	85
BRPG Bronze	BRPG Bronze	87

*IR reflectance at 1.7 μ m which is at the peak radiation frequency of burning JP-4 jet fuel.

Topcoat

The polyurethane topcoat which had been used in previous work had excellent IR transparency, which recommended it initially for this use. However, a problem arose because of excessive surface tackiness resulting in soil adhesion, and in the current work it was found that the aluminum pigment tended to rub off, indicating inadequate pigment adhesion. Several other possible topcoat materials were examined, all being urethane except for one acrylic coating. They are described in Table 4.

TABLE 4. CANDIDATE TOPCOAT MATERIALS

<u>Identification</u>	<u>Type</u>	<u>Manufacturer</u>
Super Desothane Series 800, BMS10-60, Type II	urethane	DeSoto Company
Helestatic 30CY2709	urethane	Wilmington Chemical
Helestatic 30CY2630	urethane	Wilmington Chemical
Helestatic 30JH0323	urethane	Wilmington Chemical
Solucote F/R109	flame retardant urethane	Soluol Chemical
G-cure 868/869	acrylic	General Mills

Initial evaluation of these coatings did not include the G-cure acrylic, since it was unavailable at the time the tests were run. All of the others were applied to a cotton fabric which had been primed with Viton L-31. The topcoat incorporated an equal weight of aluminum pigment.

In many of the tests, a Viton coating without topcoat was included as a control. The tests were as follows:

1. Bally Flexometer

This device exposes the specimen to severe folding and flexing. The appearance after 1000 cycles is recorded in Table 5.

TABLE 5. BALLY FLEXOMETER EVALUATION OF TOPCOATS

Substrate: 11 oz cotton, primed with Viton L-31
Topcoat: 1 coat with 1:1 pigment

<u>Topcoat Sample</u>	<u>Appearance After 1000 Cycles</u>
1. Desothane + Al #2011	severe cracking and flaking, exposed fabric
2. Helastic 30JH0323 +Al #2011	some cracking and flaking
3. Helastic 30CY2709 +Al #2011	some cracking, no flaking
4. Helastic 30CY2030 +Al #2011	some cracking and flaking
5. Solucote F/R109 +Al #2011	slight cracking and flaking
6. Viton A +BRPG	slight cracking and flaking
7. Viton A clear	no change*

*Cracking occurred at 3000 flex cycles.

2. Taber Abrasion

These tests were run using an H22 abrasive wheel under 125 gram load. The appearance after 100, 600 and 1000 cycles is recorded in Table 6. The solucote F/R109 coating had the best resistance to scuffing and abrasion, with little to choose between the others.

TABLE 6. TABER ABRASION OF TOPCOATS

H22 Wheels, 125 Gram Weight

Substrate: 11 oz cotton, primed with Viton L-31
Topcoat: 1 coat with 1:1 pigment

<u>Topcoat Sample</u>	<u>Appearance 100 Cycles</u>
1. Desothane +Al	Topcoat rubbing off, rough surface, pin holes
2. Helastic 30JH0323 +Al	Topcoat rubbing off, rough surface, pin holes
3. Helastic 30CY2709 +Al	Topcoat rubbing off, rough surface, pin holes
4. Helastic 30CY2630 +Al	Topcoat rubbing off, rough surface, pin holes
5. Solucote F/R109 +Al	Polishing, slight scratches
6. Viton A +bronze	Rough surface, dirt sticking
7. Clear Viton A	Rough surface, dirt sticking
	<u>Appearance 600 Cycles</u>
1. Desothane +Al	Slightly more severe than above
2. Helastic 30JH0323 +Al	Slightly more severe than above
3. Helastic 30CY2709 +Al	Slightly more severe than above
4. Helastic 30CY2630 +Al	Slightly more severe than above
5. Solucote F/R109 +Al	Slightly more severe than above
6. Viton A +bronze	Fabric exposed, failed
7. Clear Viton A	Fabric exposed, failed
	<u>Appearance 1000 Cycles</u>
1. Desothane +Al	Fabric exposed
2. Helastic 30JH0323 +Al	Fabric exposed
3. Helastic 30CY2709 +Al	Fabric exposed plus large holes
4. Helastic 30CY2630 +Al	More roughness, fabric just being exposed
5. Solucote F/R109 +Al	Slight roughness and scratches, more polished*

*Did not show signs of failure until over 4000 cycles.

3. Soiling and Soil Removal

Resistance to soiling and ease of soil removal are an important characteristic of the outer reflective surface of a proximity suit, for adherence of soil to the coated surface would reduce its reflectivity, and cause local "hot spots" to develop. Previous attempts to produce a reflective coated surface were deficient in this respect because of excessive surface tackiness.

The soil resistant character of the surface is largely dependent upon the characteristic of the aluminum pigment-containing urethane finish coat, though the texture of the substrate fabric may also be important, to the extent that it influences the planarity of the surface coating.

In order to evaluate relative soil resistance and cleanability of the various topcoats, the samples described in Table 5 were tested, using the standard Gentex aluminized Novatex fabric as a control.

Natural soils are varied in composition, but generally consist of oily components and solid components. Synthetic soils are usually made up of representatives of each of these classes, used either separately or mixed in various proportions. We used two soiling media, which were:

- 1) used dirty motor oil
- 2) a 15% dispersion in water of Vulcan XC-77, a finely divided carbonaceous product made by Cabot Corporation.

The procedure used for applying and removing the soils and evaluating the results was based on the standard AATCC Test Method 130-1974. Test specimens were spotted with five drops of the soil (either the oil or the carbon particle dispersion), then covered with glassine paper, a glass plate and a 5 lb weight for one minute to spread the soil evenly. After removal of the weight, plate and paper, the soiled area was exposed to the air in the laboratory for 15 minutes. Finally, the samples were washed in one of four ways:

- 1) spray with cold water from a low pressure hose
- 2) wipe with a wet sponge
- 3) spray with "Spray & Wash," an easily available emulsifying agent for home use, then use procedure (1)
- 4) spray with "Glory Foam," a carpet detergent for home use, and wipe with a wet sponge.

The specimens were then viewed under standard lighting conditions and the degree of soil removal rated on a scale of 5 (excellent), 4 (good), 3 (fair), 2 (poor) and 1 (poorest).

The results are given in Table 7.

TABLE 7. REMOVAL OF SOIL FROM URETHANE COATED FABRICS

Coating	Particulate Carbon Soil		Dirty Motor Oil	
	Cold Water Spray	Wet Sponge Wipe	Spray&Wash then water spray	Glory Foam then wet sponge
Gentex control fabric	3.0	4.0	4.0	5.0
Desothane	3.0	4.0	3.5	5.0
Helastic 30JH0323	3.0	4.0	4.0	5.0
Helastic 30CY2709	2.0	4.0	3.0	5.0
Helastic 30CY2630	2.5	3.5	3.0	5.0
Solucote F/R109	3.0	5.0	5.0	5.0

It is clear that soil can be removed easily from most of these coatings. Particulate soil can be wiped off completely with a wet sponge. Oily soil requires the use of an emulsifier like "Spray & Wash" followed by a water spray or a detergent like "Glory Foam" followed by a wet sponge wipe.

The effect of such soiling and soil removal procedures on the surface infrared reflectance will be checked, but the visual appearance is identical to the original, unsoiled surface. Of the 5 polyurethanes tested, Solucote F/R109 has the best overall properties, and Desothane and Helastic 30CY2630 were the least desirable.

IR Reflectance

Because of the good results obtained with Solucote F/R109 in the above tests, the infrared reflectance of samples using this as a topcoat was compared with the Desothane used previously. Kevlar fabric style 1110 was given one tiecoat of Viton L-31, two base coats of bronze pigmented Viton A, and finish coats consisting of one coat of clear and one coat of aluminum pigmented Desothane or Solucote. The IR reflectance measured at 1.7 μ m was 83% of the Desothane and 76% of the Solucote.

Flammability

One of the concerns about the incorporation of aluminum pigment in the topcoat was the possibility that if it were not sufficiently isolated from the Viton coat the "pyrotechnic" type of burning which results from a combination of Viton and aluminum might result. Tests were run in which a Bunsen burner flame impinged directly on the surface for 1 minute. The Solucote urethane was the only coating which incorporated a flame retardant, so all the other urethane coatings blistered and burned, while the Solucote only smoked. However, there was an excessive degree of pyrotechnic flashing with the Solucote material, which was evidenced only very mildly by the others. This, along with the somewhat reduced IR reflectance, was felt to be sufficient to eliminate the Solucote from further consideration.

The next best choice was one of the Helastic urethanes. Type 30JH0323 was selected because of its superior behavior during the coating operation.

The other two Helastic products were harder to handle in the coating operation and were stiffer in the cured coat. It proved to be a simple matter to incorporate an organic brominated plasticizer, Kromine 9050, made specifically for urethanes by Keil Chemical Division of Ferro Corporation into Helastic 30JH0323. This improved its behavior to the point where there was no blistering or flashing in the above test, but only some charring.

On the basis of all the above tests, it was decided to use the flame retardant treated Helastic 30JH0323, incorporating aluminum pigment, as the topcoat.

Foam

Considerable effort was devoted to the development of a nonflammable foam which could be used as a back coating on the fabric, to reduce heat transfer and, possibly, eliminate some of the weight and bulk contained in other layers of the proximity suit. Three types of foamed coatings were considered: Viton, silicone, and Vonar, a nonflammable neoprene foam made by Dayco Corporation for use in upholstered furniture.

(a) Vonar

Vonar is an open-cell neoprene foam supplied to us in 1/8" and 1/4" thickness. These could readily be laminated to the Kevlar fabric using a special neoprene adhesive identified as Columbia Cement Co. Inc. #2332 N/F. The lightest of these foams (11 oz/yd²) was laminated to the back of a coated Kevlar fabric and sent to Fyrepel Products, Inc. for sewing trials. Application of this foam stiffened the fabric considerably, and the foam buckled and/or cracked badly when folded or sewn. Moreover, since it was an open cell foam it had a sponge-like ability to absorb water, and would have to be protected, in the garment, by a water impermeable film. This type of foam was felt to be unusable in its currently available form.

(b) Silicone

Most silicone foams burn too easily to be satisfactory in this application. One fire retardant, two-part formulation designated as Dow Corning's 3-6548 silicone RTV foam was found, however. In trials with this material we were unable to form a stable, thin silicone foam which could be bonded to the substrate and then cured. We were unsuccessful in finding another suitable material, and the use of a silicone foam was abandoned.

(c) Viton

The only remaining type of compound which might be suitable for the purpose was a fluorocarbon, the commonest commercial example of which is the Viton which is being used for the face coating.

Foams made from high temperature resistant material such as Viton are difficult to make for reasons which involve the mechanics of foam production. In a common procedure for making a foam, a blowing agent is incorporated into the coating formulation, which is then applied to the fabric in the normal way, for example by knife coating. The coated fabric then passes into an oven where, at a suitable elevated temperature, the blowing agent produces a gas which expands the polymer by forming bubbles within its structure. For this to work successfully, the polymer modulus must be low enough at that temperature to permit easy expansion of the structure by the released gas, and the polymer viscosity must remain high enough, or the polymer must cure fast enough, to prevent the released gas from escaping through the surface. Thermoplastic polymers like polyvinyl chloride are ideal for this purpose, and PVC foams are common and easy to make. Viton is not ideal, because its modulus is not low enough at the blowing temperature of about 105°C. Because of this uncertainty we used four types of Viton in our trials, identified as Viton A, Viton A-35, Viton B-50 and Viton C-10. Formulations which produced usable foams were as given in Table 8.

TABLE 8: VITON FOAM FORMULATIONS

<u>Viton A</u>	Parts by Weight	<u>Viton A-35</u>	Parts by Weight	<u>Viton B</u>	Parts by Weight
Viton A	100	Viton A-35	100	Viton B-50	80
Maglite Y	5	Viton process aid-1	5	Viton C-10	20
Hydral 710	5	Maglite Y	5	Maglite D	5
Imsil A-10	5	Cab-o-sil M5	5	Sb ₂ O ₃	5
Fire Brake ZB	1.25	Blanc fix	10	TiO ₂	5
Cerechlor S-52	3	Sb ₂ O ₃	5	Blanc fix	5
Unicel ND	9	TiO ₂	5	Unicel ND	9.4
Aquarex NS	2.7	Unicel ND	9.4	Aquarex NS	2.7
Activator 736	1.3	Aquarex NS	2.8	Activator 736	1.3
Diak #1	0.75	Activator 736	1.4	Diak #1	0.75
		Lupersol 101	3	Lupersol 101	3
Total	133.		151.6		137.15

Mill in order using cooling
water. After milling is
completed, add

Lupersol 130 3
MEK (dry) 135
DMF (dry) 65

Cure system: Maglite Y,
Diak #1, Lupersol 130.

Blowing system: Unicel ND,
Aquarex NS, Activator 736.

After milling, dis-
solve in 2/1 MEK/DMF,
then add 1 part tri-
ethylene diamine

Calender stock onto
fabric

15 minute oven-blow
at 350°F

or 15 minute press
cure at 370°F

Our trials indicated that, while a modest expansion ratio (up to 4:1) was possible in the foaming step, an incompatibility existed between our requirements and the production of these Viton foams. We wanted a foam weighing in the vicinity of 5 oz/yd². At an expansion ratio of 4:1, this represents a thickness of only about 0.020". But because of the bubble size in the foam, it was impossible to make a foam thinner than about 0.060", weighing approximately 15 oz/yd². Attempts to make a foam thinner than this resulted in bubble collapse and a porous, unexpanded coating. In spite of this, it might be worthwhile to use a 15 oz/yd² foamed back coating if the thermal insulation it provided permitted a reduction of at least this weight in another part of the suit.

The results of measurements of heat transfer rate under a radiant heat flux of 1.2 cal/cm²/sec and a flame impingement source at a flux of 1.3 cal/cm²/sec for a number of different fabric and coating combinations are given in Table 9, and are plotted in Figure 1.

TABLE 9: HEAT TRANSFER CHARACTERISTICS

Sample No. and Material	Weight (oz/yd ²)	Time for 50°C Rise Behind Sample (sec)	
		Flame	Radiant
		Impingement 1.3 cal/ cm ² /sec	Heat 1.3 cal/ cm ² /sec
1 3M Aluminized Novatex	15.5	7.1	18.6
2 Bronze Viton/Novatex	16.9	8.7	10.3
3 Bronze Viton/Kevlar	9.6	5.3	7.1
4 1 layer uncoated Kevlar	6.7	6.0	6.0
5 2 layers uncoated Kevlar	13.4	8.2	7.9
6 3 layers uncoated Kevlar	20.0	10.7	12.5
7 Kevlar/Viton A35 foam	21.3	18.9	----
8 Kevlar/Viton B50 foam	19.2	18.6	14.1
9 Al acrylic/bronze Viton/Kevlar	10.1	7.7	8.3
10 Clear acrylic/Al acrylic/bronze Viton/Kevlar/Viton A35 foam	25.3	18.2	-----*
11 Clear acrylic/Al acrylic/bronze Viton/Kevlar/Viton B50 foam	19.2	18.3	----
12 Uncoated Novatex	14.1	----	7.2

*The acrylic topcoat used in this fabric was not flame retardant. Consequently this surface coat ignited after 10 seconds of exposure. If ignition had not occurred, this time would have been approximately 19 seconds.

For the most part, the heat transfer rate when exposed to radiant heat is somewhat less than when exposed to an impinging flame. This is due to the high thermal conductivity characteristics of the metal in the coating, which are important when the exposure involves flame contact, but of less importance when the heat flux is pure radiation. The effect is most apparent in the aluminized Novatex (Sample 1), for which the heat transfer is essentially the same as that for uncoated Novatex (Sample 12) when the source is an impinging flame, but much lower when the source is radiant. Note that there is essentially no difference between the results for the two sources when the sample is uncoated (Samples 4, 5 and 6).

The line which is drawn in Figure 1 through the results for Samples 4, 5 and 6 can be taken to represent the effects of changes in mass on heat transfer. Most of the results for the coated samples fall close to this line, indicating that the most important contribution of the coating is an increase in mass. The exceptions to this general observation are the aluminized Novatex when the source is radiant, and the samples having a foam backing, in which the foam contributes somewhat better insulation than a solid material of its same mass, as would be expected. The good behavior of the metallized surface under radiant heat (not under flame impingement) results, no doubt, from its extremely high reflectance, which can only be obtained from a pure metal surface. However, this type of surface exhibits a poor wear resistance

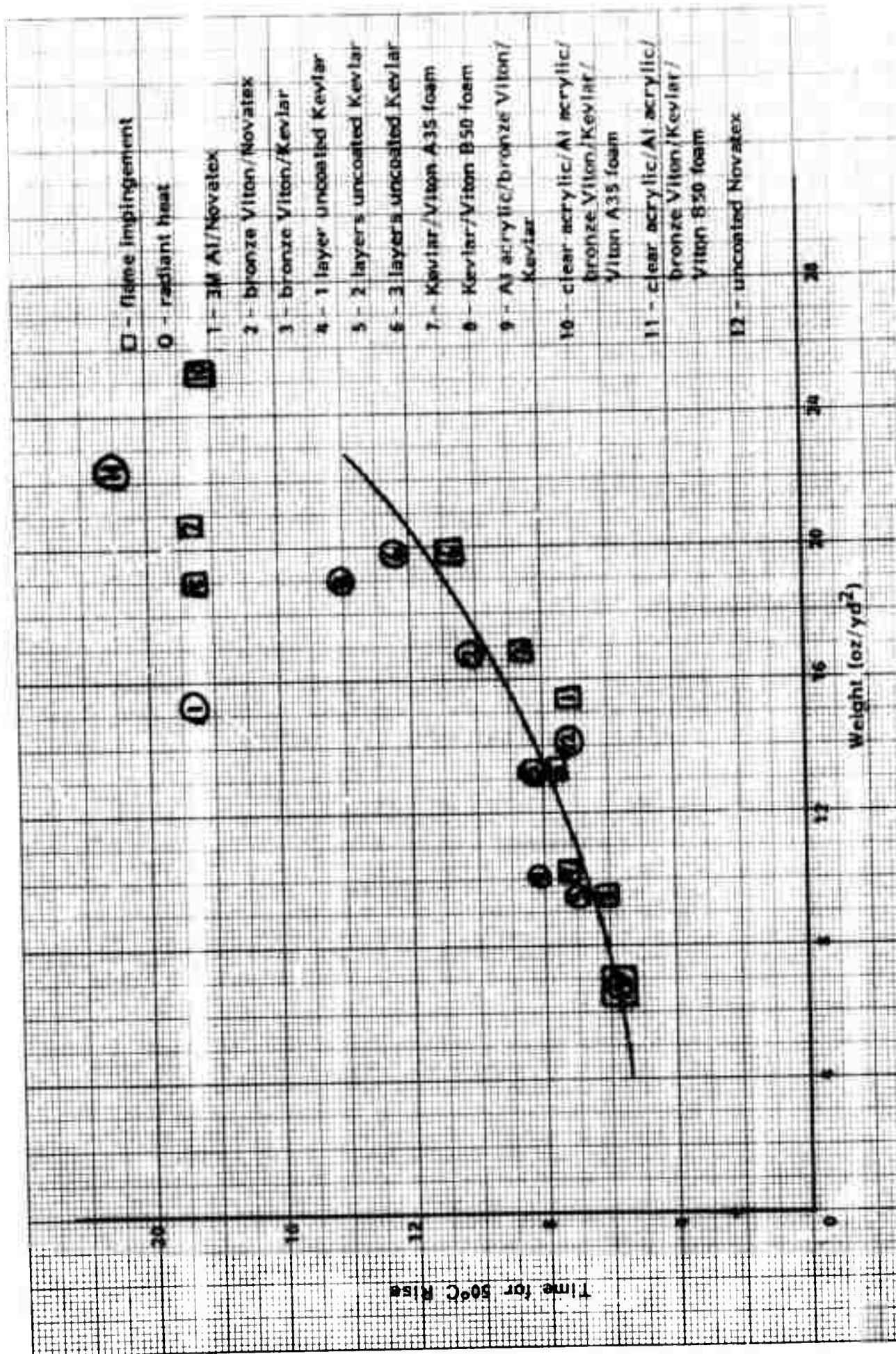


Figure 1. Heat Transfer Rates for Various Coatings

which it is the purpose of this contract to attempt to improve. The foam layer was used to decrease the rate of heat transfer, as it has done. But in order not to add too much weight, the foam had to be made as thin as possible, and the 10-12 oz/yd² of foam represents the smallest amount which could be made to foam. Less than that amount produced a porous surface but not an expanded film. This minimum foam layer is not as low density, however, as might be achieved with a thicker foam. Moreover, it has significantly stiffened the material to the point of marginal acceptability. The use of the foam layer must be decided, therefore, in light of both the advantages and disadvantages outlined above.

Effect of Surface Reflectivity on Heat Transfer Rate

In FRL's final report under contract F33615-75-C-5168, now summarized in AFML-TR-77-72, "The Transient Thermomechanical Response of Protective Fabrics to Radiant Heat," an equation was derived which gives the relationship between time t (sec) and specimen temperature T_2 (°K) when exposed to a radiant source operating at temperature T_1 (°K). For unilateral exposure, and no loss in heat from the back of the specimen, this relationship is given by:

$$t = \frac{W/A}{4\sigma} \left[\frac{1}{\epsilon_1(T_1)} + \frac{1}{\epsilon_2(T_2)} - 1 \right] \left\{ \frac{a}{T_1^2} \ln \left[\frac{T_1^2 - T_0^2}{T_1^2 + T_0^2} \cdot \frac{T_1^2 + T_2^2}{T_1^2 - T_2^2} \right] + \frac{b}{T_1^3} \ln \left[\frac{T_1 - T_0}{T_1 + T_0} \cdot \frac{T_1 + T_2}{T_1 - T_2} \right] + \frac{2b}{T_1^3} \left[\tan^{-1} \left(\frac{T_2}{T_1} \right) - \tan^{-1} \left(\frac{T_0}{T_1} \right) \right] \right\}$$

In this equation t , T_1 and T_2 are defined as above, and T_0 (°K) is the original specimen temperature; W/A (gm/cm²) is the specimen weight per unit of effective area (the projected area of the solid material); σ is the Stefan-Boltzmann constant, 1.354×10^{-12} cal/cm²/sec °K⁴; $\epsilon_1(T_1)$ is the heater emissivity at temperature T_1 ; $\epsilon_2(T_2)$ is the specimen emissivity at temperature T_2 ; a and b are constants having values of 0.00053 cal/gm/°K² and 0.15 cal/gm/°K respectively. Values for the heater emissivity for a range of heater temperatures are given in the report referred to above. Values for fabric emissivity can be inferred from data given in a paper by J. Quintiere entitled "Radiative Characteristics of Firefighters' Coat Fabrics" (Fire Technology, Vol. 10, No. 2, May 1974, 153-161), and the gray body assumption that fabric emissivity and absorptivity are equal. This indicates that fabric emissivities lie in the range 0.7-0.9 throughout the spectrum emitted by a black body source at 980°C.

A value of 0.7 for a source temperature of 1000°C seems reasonable. This corresponds to a reflectance of 0.3 for an uncoated fabric.

These values can be substituted into the above equation to calculate a time-temperature curve for fabric of any desired W/A . This, however, is not the characteristics of interest to our current work. Rather, we are interested in the influence on the rate of heating of changing the fabric reflectance by application of a coating. This influence is depicted in Figure 2, in

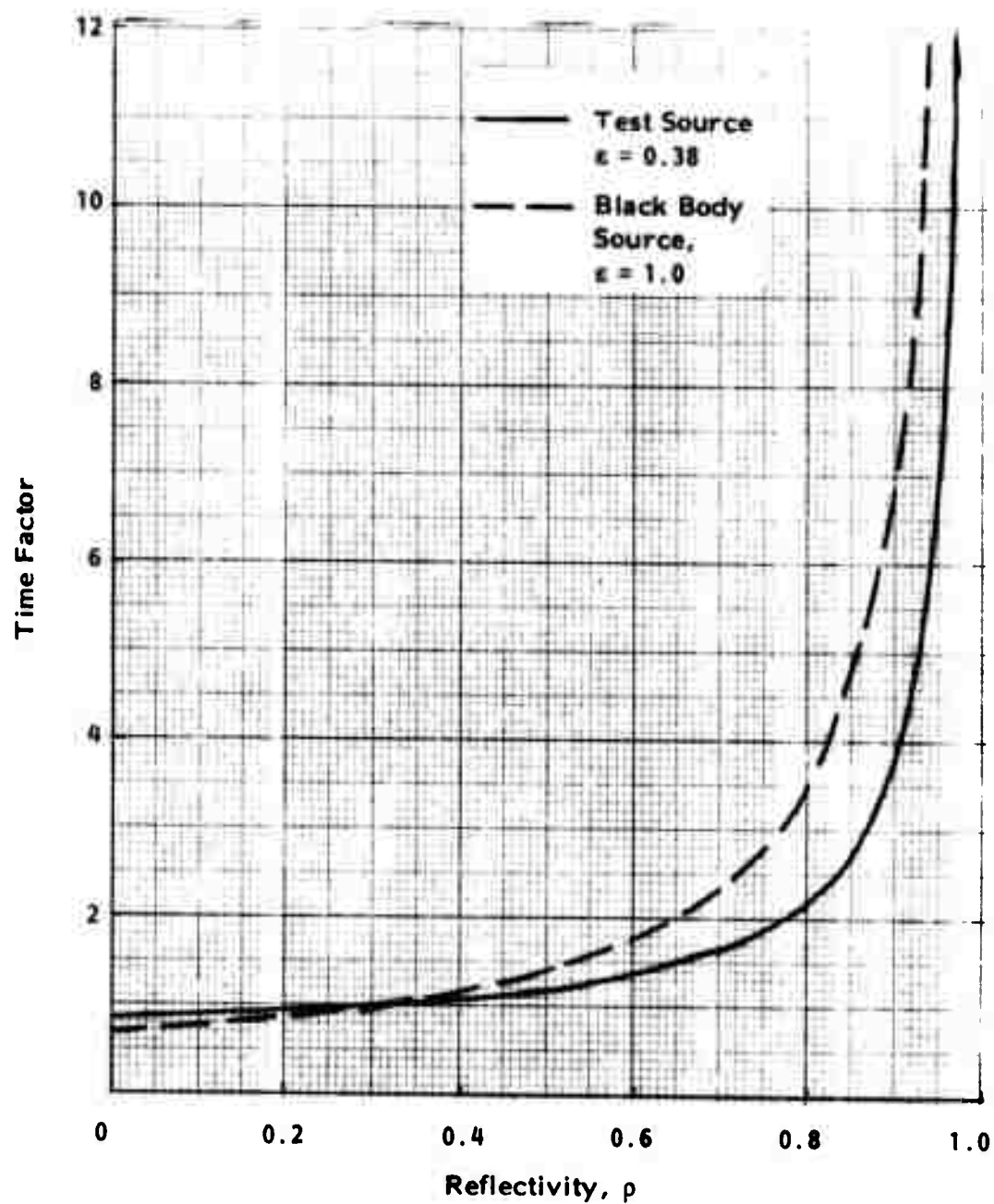


Figure 2. Effect of Surface Reflectivity on Heat Transfer Rate

which is a time value of 1 arbitrarily assigned to a reflectivity of 0.3. The two curves are drawn for source emissivities of 0.38, representing the quartz panels used in our tests, and 1.0 for a black body represented by a large fire or a hot, sooty surface.

The results are very revealing, indicating that reflectivity has little effect until it is higher than about 0.8. It is apparent that the 0.95 value which is typical of a clean aluminum surface is many times more effective than the values of 0.7-0.8 which our fabrics have given. Indeed, our attempts to increase reflectivity even by 0.05 in this range were obviously of little real value. It seems that if one must have better durability and retention of reflectivity than can be obtained from an unprotected aluminum surface, the garment design will have to be adapted to accommodate surface reflectivities less than 0.8.

The effectiveness of our coating can be assessed by comparing with reflectivities for coated and uncoated fabrics given in Quintiere's paper referred to above, and in a paper by R. V. Dunkle, F. Ehrenburg and J. T. Gier entitled "Spectral Characteristics of Fabrics from 1 to 23 Microns" (Transactions of the ASME, February 1960, 64-70). Some figures from these papers have been reproduced herein. Figures 3 and 4 come from Dunkle et al, and Figure 5 from Quintiere. Within the range of wavelengths of particular interest, 0-10 μm , uncoated fabrics all show low reflectance around 3 μm and higher reflectance in the 4-7 μm region. Application of a bright aluminum coating raises the reflectance to a uniform 0.95+ (curve 14 of Figure 5). A vacuum deposited aluminum coating, which is grey in color, gives a reflectance which varies between about 0.2 and 0.4 (curve 15, Figure 5). Miliun, an aluminum pigment dispersed in a resin, gives a fairly uniform reflectance of about 0.4. Our topcoat, aluminum in polyurethane, gives a value which ranges between 0.7 and 0.85, or lower by approximately 0.1 when protected by a clear finish coat of polyurethane. An uncoated, unpigmented Viton coating gave a value of 0.7.

This analysis has given us a better insight into the function of the coating, and specifically points up the fact that at the levels of reflectivity which are possible with a resin-protected and, therefore, more durable aluminum surface, marginal increases in reflectivity (less than say 0.1) are relatively ineffective. Indeed, the most efficient way of improving the performance of a proximity suit probably lies in a judicious distribution of mass. (Note that time to temperature in the above equation is almost proportional to mass per unit projected area (W/A) since the first term in the expression is much larger in magnitude than the other two terms.)

The coated fabric which is being considered for use in proximity suits consisted of the following:

- 1) A 6.7 oz/yd² Kevlar base fabric.
- 2) A prime coat of clear Viton B, followed by 2 coats of a 1:1 (by weight) mixture of Viton A and BRPG bronze pigment. Total weight approximately 3 oz/yd².
- 3) A topcoat of flame resistant polyurethane containing aluminum pigment. Total weight approximately 3 oz/yd².
- 4) A finish coat of clear urethane, weight 0.5-1.0 oz/yd².

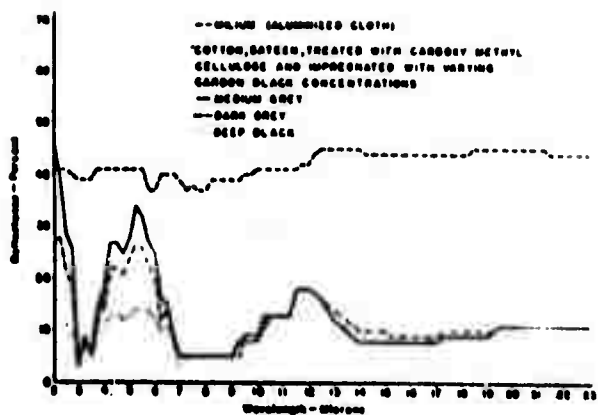


Figure 3. Reflectance, Millum and Carbon-Impregnated

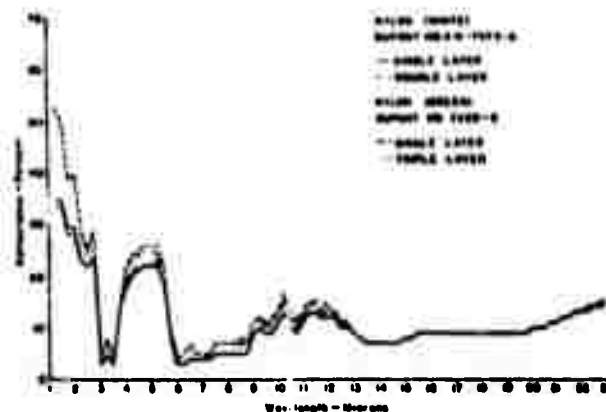
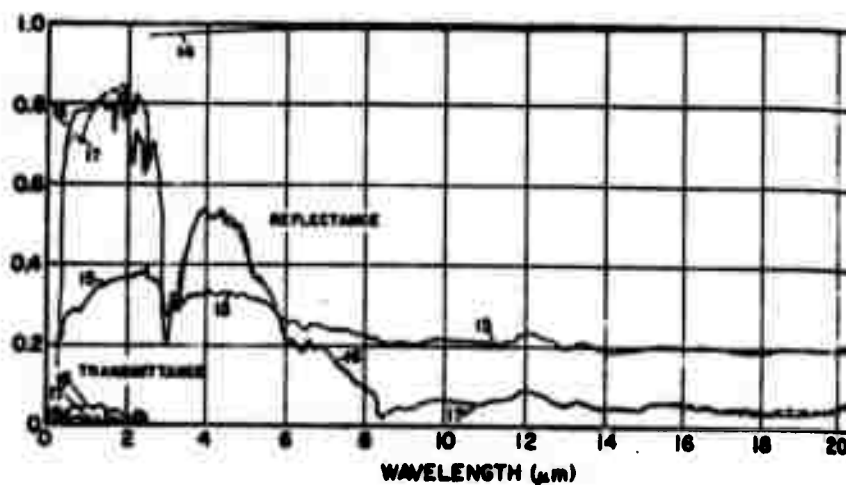


Figure 4. Reflectance, Nylon



Identification number	Material description	Color	Weight (oz/yd ²)
14	Aluminized modined aromatic polyamide	aluminum	4.2
16	Glass/aro. polyamide with vacuum deposited aluminized face	grey	17.1
16	Glass/aro. polyamide	white	17.6
17	Glass/aro. polyamide Al film with scrim face	white	18.1

Figure 5. Spectral Reflectance and Transmittance for Aluminized Fabrics

It now seems that the bronze pigment, whose function was to raise the reflectivity of the Viton coating from 0.7 to approximately 0.85, is of little value because of the effectiveness of the aluminum-filled topcoat. Accordingly, hand samples were prepared in which the bronze pigment was left out of the Viton, along with one including the bronze pigment for direct comparison. Specifically, these samples were as follows:

- Sample 13: 6.7 oz/yd² Kevlar fabric
Clear Viton B tiecoat
2 coats Viton A/bronze pigment
1 coat NF polyurethane/aluminum pigment
1 coat clear NF polyurethane
- Sample 14: 6.7 oz/yd² Kevlar fabric
Clear Viton B tiecoat
2 coats clear Viton A
1 coat NF polyurethane/aluminum pigment
1 coat clear NF polyurethane
- Sample 15: 6.7 oz/yd² Kevlar fabric
Clear Viton B tiecoat
2 coats clear Viton A
2 coats NF polyurethane/aluminum pigment.

The results of heat transfer measurements using flame impingement and radiant heat sources are plotted in Figure 6, along with repeat measurements on some of the reference materials used in the measurements described in Table 9.

It is apparent from these results that the presence of the bronze pigment in the Viton does not contribute to the overall heat transfer characteristics of the material. The difference in heat transfer rate between Samples 13 and 14 cannot be attributed to the presence or absence of bronze in the Viton, but rather to the effectiveness with which the single coat of aluminized polyurethane has covered the surface. It is apparent that 2 coats of pigmented urethane are necessary to ensure good cover, as indicated by the results for Sample 15. The very high value obtained for 3M aluminized Novatex (Sample 1), which was 32 as compared to 19 reported previously, is only an indication that, because of the steepness of the reflectance/time-to-temperature curve for these high reflectance levels (Figure 2) measurements cannot be expected to be closely reproducible.

Final Design

As a result of these measurements, the desirability of including the bronze pigment in the Viton, and applying a Viton foam to the coated fabric was discussed by representatives from FRL with the Project Engineer and with a fire clothing manufacturer.

(a) Use of a bronze pigment

Our analysis indicated that the bronze pigment contributed little to the overall performance except mass, and its inclusion implied additional cost, increased thermal conductivity, increased fabric stiffness, and the possibility that its mass might better be present in a different

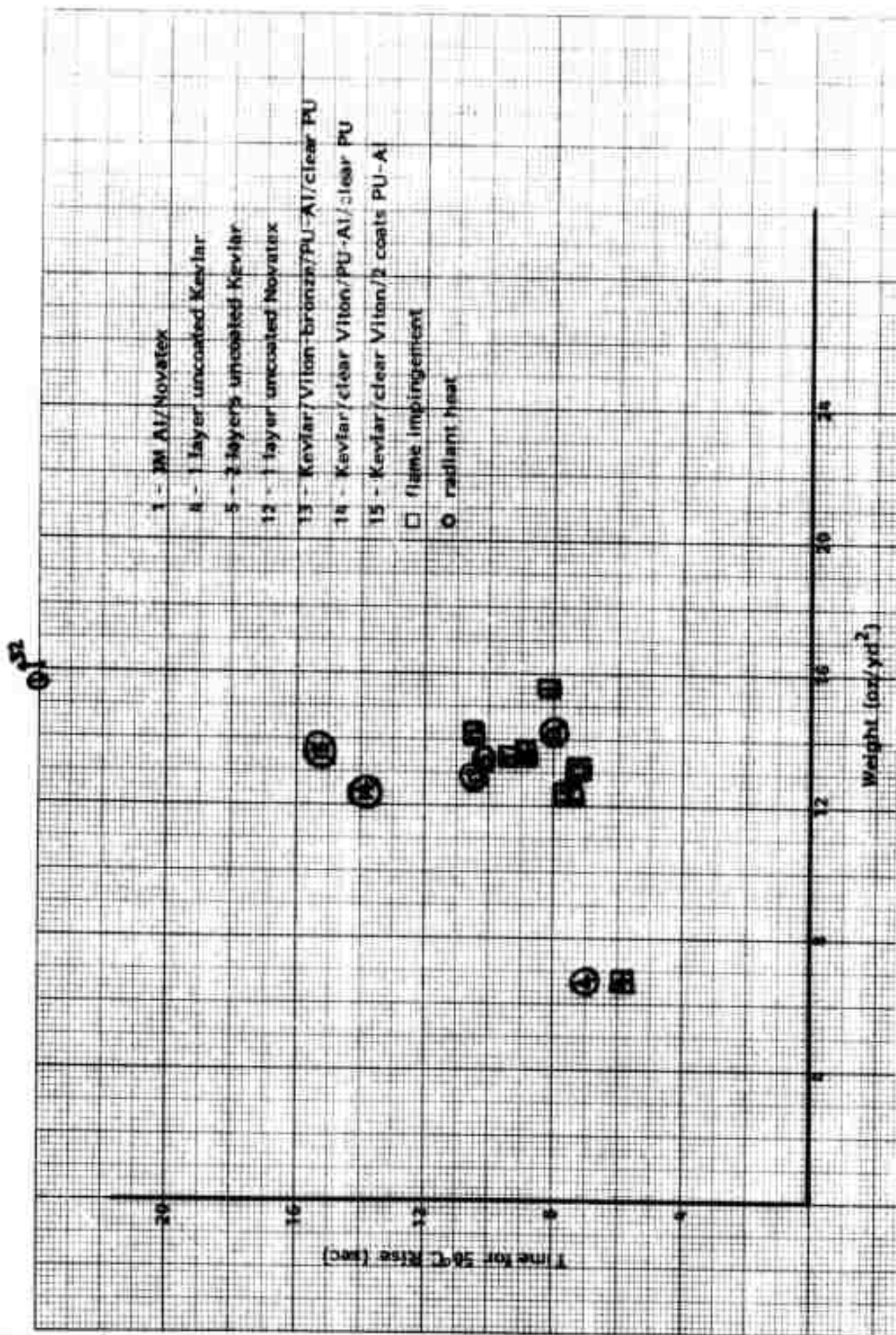


Figure 6. Heat Transfer Rates for Various Coatings Subjected to Flame Impingement or Radiant Heat Sources

material elsewhere in the garment assembly. Nevertheless, it was felt that to omit it on the basis of an analysis of unproved practacility was too great a risk at the present time, and it should be included. Moreover, if any damage to the topcoat occurred, the bronze pigment could be expected to function adequately in place of the aluminum.

Use of the foam, however, was harder to justify. If a lower density foam could be made, it would overcome some of the questionable features of the current foam, and could probably be a valuable component in the assembly. The matter of producing a better Viton foam was discussed with the FNF division of the Du Pont Company, and our approach to foam development and degree of success was described. They expressed an interest in working on the development of an improved foam, but their timing was such that this could not be pursued within the terms of the present contract. Therefore it was decided to abandon the use of the foam for the time being, but it remains an interesting possibility which should be pursued in future work.

The final design was as follows:

Base fabric - 6.7 oz/yd² Kevlar 29, Style 718

Prime coat - L-31, Viton B latex formulation

Base coat - at least 2 coats of the bronze pigmented Viton A formulation

Topcoat - at least 2 coats of aluminum pigmented, flame resistant polyurethane Helastic 30JH0323

Finish coat - one coat clear, flame resistant polyurethane Helastic 30JH0323.

Production of Deliverable Yardage

Contract requirements included the production of approximately 400 yards of material coated under commercial plant conditions.

Arrangements were made to coat the required yardage of Kevlar fabric in a large coating plant which FRL has worked with in the past. The production trial was unsuccessful and no usable material was produced. This was primarily due to inexperience in handling Kevlar fabric, aggravated by the poor quality of the fabric which was not designed specifically for coating use. Much was learned however; especially that a specific type of commercial coating equipment better adapted to handling inextensible fabric would have to be utilized.

After some additional development work and a laboratory trial run, the decision was made to have Reeves Bros., Inc., Rutherfordton, NC, attempt to accomplish the coating of the Kevlar fabric.

Four hundred yards of Kevlar fabric were coated with the formulations developed during this program as follows:

- 1) 30 yards used and not recovered in a preliminary run to establish operating procedures and the proper coating formulation viscosity.

- 2) 30 yards used to check out process changes made from information obtained from preliminary run. 27 yards of this material recovered.
- 3) 340 yards production run.

During part (1), the preliminary run, severe blistering of the aluminum top-coat occurred. In order to reduce or eliminate the blistering the coating method was changed from knife-over-roll to scrape coating (knife-over-slot), the coating formulation viscosity was reduced and oven drying temperature was reduced (drying time therefore was increased).

It was further noted that three bronze base coats were needed to achieve a smooth surface in the coated fabric. The final 340 yards were made according to the finalized processing conditions as reported in the appendix. No calendaring was necessary and it was easy to keep total coated fabric weight less than 15 oz/yd². No fabric handling problems arose, though the presence of weaving defects in the base fabric made it impossible to produce a completely defect-free coated fabric.

The cost per yard of coating 400 yards of Kevlar 29 fabric, Style 718 with the coating formulation as developed in this contract and as listed in the appendix has been calculated, and is shown in Table 10. These costs are based on the actual production run made by Reeves Bros. and include fabric and all material costs based on current (1977) prices, and actual plant operation costs. The total cost per yard to produce 360 yards of fabric, 48" nominal coated width is \$19.43 per linear yard. Reeves Bros. Inc., would quote today (1977) a price of \$24.43 per linear yard for 360 yards delivered, which would allow them a contingency and profit of \$5.00 per yard based on the above cost estimate.

TABLE 10: MATERIAL AND COATING COSTS -
FIREFIGHTERS' PROTECTIVE CLOTHING OUTER FABRIC

Basis: 360 linear yards finished goods, 90% fabric yield.

<u>Item</u>	<u>Quantity Required</u>	<u>Unit Cost</u>	<u>Total Cost</u>
1. <u>Prime Coat</u>			
Viton L-31	88 lb (dry)	\$10.00/lb	\$ 880.00
ZnO dispersion	1 gal	20.00/gal	20.00
Diak #3	3 lb	8.25/lb	24.75
Misc chemical	1 lb	2.25/lb	2.25
2. <u>Base Coat</u>			
Viton A	60 lb	12.00/lb	720.00
BRPG pigment	60 lb	2.79/lb	167.40
Cure system	1-1/2 lb	26.97/lb	40.46
Solvents			
MEK	100 lb	0.65/lb	65.00
DMF	50 lb	0.75/lb	37.50
Toluene	45 lb	0.60/lb	27.00

TABLE 10: MATERIAL AND COATING COSTS -
FIREFIGHTERS' PROTECTIVE CLOTHING OUTER FABRIC (cont)

Basis: 360 linear yards finished goods, 90% fabric yield.

<u>Item</u>	<u>Quantity Required</u>	<u>Unit Cost</u>	<u>Total Cost</u>
3. <u>Top Coat</u>			
Helastic 30JH0323	80 lb	1.38/lb	110.40
Kromine 9050	2.4 lb	0.80 lb	1.92
#2011 aluminum	72 lb	2.08/lb	149.75
Solvent MEK	8 lb	0.65/lb	5.20
4. <u>Clear Over Coat</u>			
Helastic 30JH0323	40 lb	1.38/lb	55.20
Kromine 9050	1.2 lb	0.86/lb	1.03
5. Total Cost of Coating Materials		6.41/yd	\$ 2,307.86
6. Kevlar 29 Fabric Style 718	400 yd	7.95/yd	3,180.00
	<u>Cost/Linear Yard</u>		<u>Total Cost</u>
7. Total Material Cost	15.24		\$ 5,487.86
8. Processing Cost	3.00		1,080.00
9. Finishing, Packing, Shipping	1.19		430.00
10. Total Cost	\$19.43		\$ 6,997.86

Conclusion

The Viton-based coating processed well when applied by a commercial coater experienced in handling Kevlar fabric. There remains, however, the relatively poor quality of commercial fabric woven from Kevlar filament yarn. Since the decision was made to use this fabric, staple Kevlar has become a standard commercial item. It is likely that a fabric made from staple Kevlar would be more uniform and cheaper than the filament fabric made in the current contract, and would produce a better coated fabric, and a garment at least as serviceable as one based on the filament fabric. Any further work, then, should include a study of the following:

- (a) Kevlar staple fabric in place of Kevlar filament fabric
- (b) Possible omission of the bronze pigment from the Viton coating
- (c) Development of a lower density, more flexible Viton foam for use as a back coating
- (d) Any other problems which may arise in the wear test to be carried out using the fabric produced in this contract.

APPENDIX

PROCESSING OF THE COATED KEVLAR FABRIC

Date: September 27-29, 1977

Place: Reeves Brothers, Inc., Vulcan Plant
P. O. Box 671
Buena Vista, VA 25516

For Reeves Bros: Director - Richard M. Kerr
Chief Chemist - Richard Lugar
Trial Supervisor - Ralph F. Tomlin
Plant Manager - Don Armstrong

For FRL: Supervisor - Robert E. Erlandson

A. COATING FORMULATIONS

	<u>Parts by Weight</u>
1. <u>Prime Coat (60% total solids)</u>	
Viton L-31 (63%)	159
ZnO dispersion (60%)	17
Diak #3 dispersion (33%)	9.1
Acrysol GS (25%)	4
2. <u>Base Coat (48% total solids)</u>	
Viton A (35%)*	286
BRPG pigment	100
Triethylenediamine	1
Benzoyl peroxide	1
MEK	30
3. <u>Aluminum Top Coat (35% total solids)</u>	
Helastic 30JH0323 (30%)	333
Kromine 9050	10
Aluminum #2011 pigment	90
50/50 MEK/toluene	110
4. <u>Clear Over Coat (30% total solids)</u>	
Helastic 30JH0323 (30%)	330
Kromine 9050	10

B. BASE FABRIC (400 yards)

J. P. Stevens, Style 718, Kevlar 29, 50-1/2 inches wide, scoured,
1500 denier 16-1/2 x 16-1/2.

*Viton A is dissolved in a 2/1 blend of MEK/DMF.

C. PROCESSING

A single station, knife coating line, employing a steam heated hot-air oven with automatic tension control and edge guiding was used to process the material.

A 1/8 inch round-edge knife over an 8 inch, 85 durometer rubber covered roll was used to apply the prime coat and base coat. A 1/8 inch, round-edge, floating knife or scrape coater was used to coat the aluminum top coats and clear over coat.

After the prime coat was applied and also after all the base coat was applied the coated fabric was cured in a festoon dryer. The following processing procedure was followed.

<u>Step</u>	<u>Processing Specifications</u>	<u>Weight (oz/yd²)</u>
1	Coating pass #1 - spread coat side 1, L-31 prime coat, 5 yd/min at maximum heat (90 psi steam) fabric coating	6.7 1.8
2	Festoon heater cure - 15 min at 175°F, 15 min at 230°F, 30 min at 300°F	
3	Rewind	
4	Coating pass #2 - spread coat side 1, base coat, 10 yd/min	0.8
5	Coating pass #3 - spread coat side 1, base coat, 10 yd/min	0.7
6	Coating pass #4 - spread coat side 1, base coat, 10 yd/min	0.7
	NOTE: passes 2-4 use medium heat (60 psi steam)	
7	Festoon heater cure - 15 min at 175°F, 15 min at 200°F, 15 min at 230°F, 30 min at 300°F	
8	Rewind	
9	Coating pass #5 - scrape coat side 1, aluminum topcoat, 5 yd/min	0.6
10	Coating pass #6 - dry pass, 5 yd/min	
11	Coating pass #7 - scrape coat side 1, aluminum topcoat, 5 yd/min	0.5
12	Coating pass #8 - scrape coat side 1, aluminum topcoat, 5 yd/inch	0.4

<u>Step</u>	<u>Processing Specifications</u>	Weight (oz/yd ²)
13	Coating pass #9 - scrape coat side 1, clear over coat, 5 yd/inch	0.4
	NOTE: passes 5-9 use minimum heat (front 10 psi, back 40 psi, bottom 60 psi)	
14	Rewind, dry out	
15	Inspection	
16	Packing	
	Total Weight	12.6
	Total Crossing	5.9

INITIAL DISTRIBUTION

AFCEC/DOZ	1	NASA-Johnson Space Center	1
Det 1 (CEEDO) ADTC/CNS	1	Naval Plant Rep Officer	1
Fabric Research Laboratory	5	Naval Supply Sys Command	1
AFRPL/RPMCH	1	Office of Naval Research	1
AFRPL/RPPR	1	Officer in Charge/Navy Clothing	1
AFSWC (Technical Library)	1	and Textile Research	1
Air University Library	1	Ordnance Materials Research	1
Army Materials Research Agency	1	Office	1
Chief, Naval Air System	1	Redstone Scientific Inf Center	1
Chief of Naval Material	1	SAALC/MMEMB	1
Chief of Naval Research	1	NADC (AML)	1
Commander, Naval Ship Sys	1	NASA/Crew Sys Div	1
Commander, US Naval Ordnance	1	NASA/Ames Research Center	1
Commander, US Naval Ordnance	1	NASA/Marshall Space Flight Ctr	1
Test Station	1	NASA/Crew Sys Div EC-7	1
Commander, Picatinny Arsenal	1	NASA/Lewis Research Center	1
Chief, Naval Research	1	NASA/Langley Research Center	1
Commander, Naval Air System	1	AFML/MX	1
Command Headquarters	1	ASD/ENESP	1
Commanding Officer, US Army	1	ASD/OIP	1
Mobility Equip R&D Center	1	HQ USAF/AFROPS	1
Defense Documentation Center	2	AFAL/TSR	1
Dept of Army, Office of Chief	1	AFAL/TSR/M	1
for R&D	1	AFML/MBC	15
Dept of the Navy, Naval Weapons	1	AFML/MB	1
Support Activity	1	SAMSO/YAPT (AFML/DY)	1
Director of Flight Safety	1	US Army/AMXDO-DCC	1
Research AFCFS-D-2	1	US Army Natick Labs/C&O	1
Director of Naval Research Lab	1	Materials Div	1
Director, Aero Materials Dep	1	US Army Research Office	1
FAA/NAFC (NA-542)	1	(DURHA)	1
Frankford Arsenal C/O 1421	1	US Dept of Agriculture Forest	1
HQ USA Army Chemical Corps	1	Products Lab	1
HQ USAF/AFRDDG	1	Det 1 ADTC/PRT	1
HQ Dept of Army, Office of R&D	1		
HQ USAF/AFRSTC	1		
Material Div/OSD (DDR&E)	1		
US Dept of Agriculture R&D	1		
US Dept of Commerce, Bureau	1		
of Standards	1		
US Dept of the Navy/Code	1		
RRMA-32	1		
US Naval Ordnance Lab/	1		
Nonmetallic Materials Div	1		
US Naval Ordnance Lab/Code W	1		
US Army Natick Labs/Clothing	1		
and Life Support Equip Lab	1		
Watervliet Arsenal	1		
Naval Post Graduate School	1		
Commander, Naval Ordnance Lab	1		